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COMPLETE SPECIFICATION

Process for the production of Electrostatographic Toner Particles

We, RANK XEROX LIMITED, a British Company of 37/41 Mortimer Street, London, W.1, England, do hereby declare the invention, for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:

This invention relates in general to electrostatography and in particular to an electrostatographic toner particles and a method for their production.

In the past, these toners have generally been prepared by thoroughly mixing components comprising heat softened resins and coloring materials in a device such as a Banbury mixer, followed by rubber milling to uniformly disperse the colorant throughout the resin. This blend is then allowed to solidify by cooling and subsequently broken up into relatively large particles in a grinder such as a Fitz Mill. After this rough grinding, the material is broken down further to the final particle size by jet pulverization.

This final reduction produces a powdered material having a relatively irregular shape and particle size. Although the average particle size of the toner produced generally ranges between 5 and 10 microns, individual particles ranging from sub-micron size to above 20 microns are generally produced in substantial quantities.

It is obvious then that an inherent disadvantage of the above process is that control of particle size with any degree of accuracy is extremely difficult. The conventional techniques of toner production also

have the disadvantage of producing particles of non-uniform and irregular geometry. This non-uniformity of size and shape of the particles tends to result in the production of non-uniform triboelectric relationships between the particles and their carriers if a carrier is used, and also, tends to produce non-uniform relationship between the particles and the electrostatic field being developed. Since the range of particle sizes produced by prior art techniques is relatively wide, even a toner with an average particle size which is quite low may be unsatisfactory for micro imaging or other high resolution imaging techniques because of resolution of the final image produced is limited by the largest particle size included within the developing mixture. If a particle is larger than the distance between two lines which are resolved in the latent electrostatic image, this particle will ruin the resolution between these lines when it is used to develop one of them. In addition to the aforementioned disadvantages, prior art techniques of toner production imposed certain limitations upon the material selected for the toner because the resin-pigment dispersion must be sufficiently friable so that it can be pulverized at an economically feasible rate of production. The problem which arises from this requirement is that when the resin pigment dispersion is sufficiently friable for high speed pulverizing, it tends to form an even wider range of particle sizes during pulverization and is frequently subject to further pulverization or powdering when it is employed for developing in xero-

[Price 4s. 6d.]

graphic copying apparatus, or because of movement in the developing section of the apparatus. The prior art toner production technique is also batch-type operation rather than a continuous operation so that its use is uneconomical and undesirable from a commercial viewpoint.

The present invention provides a method of preparing electrostatographic toner particles comprising forming a plurality of surfactant micelles in an aqueous medium and providing in said aqueous medium, in the presence of a water soluble polymerization catalyst, a sufficient supply of at least one ethylenically unsaturated monomer to form an aqueous suspension of monodisperse, surfactant encapsulated, polymer particles having an average size from about 0.01 to about 50 microns in diameter in said aqueous medium by preferential diffusion of monomer to the smallest of said polymer particles during polymerization, adding to said aqueous suspension a dye capable of colouring said polymer particles therein and then separating said particles from said aqueous suspension.

The polymer particles making up the resin portion of the toner are first grown to the desired size in such a manner that a monodisperse size distribution is produced and then these particles are colored to produce the final toner. Polymer growth is carried out in a liquid medium and the polymer particles may be colored directly therein by the addition of a colorant which is soluble in that liquid followed by subsequent drying of the particles as by spray drying or other suitable techniques. For the purposes of this specification and the appended claims, "monodisperse" is defined as referring to a particle system wherein the majority of particles have a diameter within plus or minus 10% of the mean particle diameter. The growth of the polymer particles to the desired size is preferably accomplished by a carefully controlled form of emulsion polymerization. As is well known to those familiar with emulsion polymerization, broadly, this technique involves mixing water, surfactant, initiator, and the monomeric precursor of the polymer to be formed. When this mixing occurs, the surfactant molecules line up in the water phase in orderly groups known as micelles. Some monomer is dissolved in these micelles. In addition, relatively large monomer droplets are stabilized by surfactant action and most frequently also by agitation of the mixture during the polymerization process. These monomer droplets may be thought of as reservoirs of monomer which are distributed later as needed in the polymerization reaction. When initiator diffusing through the water contacts monomer, it can initiate polymerization, assuming the system is at a suit-

able temperature. As polymerization proceeds in the micelle, the monomer therein is used up. The depleted monomer is replenished by diffusion from the large monomer droplets and gradually the micelles become transformed into monomer swollen polymer particles. Both during and after polymerization the polymer particles are surfactant encapsulated, i.e., the surfactant is concentrated at the water-polymer interface, thereby preventing particle coalescence or coagulation. If, as in conventional emulsion polymerization, a relatively large amount of surfactant is initially employed or added with additional monomer during the polymerization process, a large number of micelles are formed and the polymerization goes to completion at a relatively rapid rate because each of these micelles constitutes a locus of polymerization which is "fed" by diffusion from the monomer reservoir droplets. Since a large number of micelles are provided with this technique, a large number of very small polymer particles are formed at a rapid rate and the monomer is soon depleted from the system. In any emulsion polymerization system of this type, the monomer tends to diffuse to the micelles at a rate which is inversely proportional to their diameter so that there is a tendency in emulsion polymerization for all of the polymer particles finally produced to be of uniform size. This generally does not occur in conventional emulsion polymerization because there is not enough monomer supplied to allow the particles to grow to uniform size since the large number of micelles use up the initially supplied monomer at a relatively rapid rate before the size related selective diffusion effect described above can come into full play. This difficulty can however be overcome by a technique which involves growing what may be thought of as relatively large polymer particles when compared with the polymer particles produced in ordinary emulsion polymerization. In this technique, the polymer particles are grown to the desired size, say, for example, two microns by initially supplying an amount of surfactant capable of forming the required number of micelles and then providing sufficient monomer so that the particles can grow to the desired size according to the selective diffusion phenomena described above. The monomer may be provided by initially supplying extremely large reservoirs of monomer either emulsified in the aqueous phase if a liquid monomer is used or overlying the aqueous phase if a gaseous monomer such as butadiene is used or by seeding the system with additional monomer from time to time so that when polymerization of a first batch of monomer is complete, additional monomer is supplied to allow for continued growth of the par-

ticles or by continuously adding monomer during the polymerization in sufficient quantity to achieve the desired final particle size.

Although the above-described polymerization technique allows for the growth of polymer particles within the size range desirable for electrostatographic toners which includes a preferred range of from about 0.01 to about 10 microns in diameter and a more preferred range of 0.1 to 5 microns, it may, on occasion, be desirable to produce larger particles ranging up even as high as 50 microns. When such larger particles are produced without the addition of any surfactant after the first batch of surfactant is placed in the system, it will generally be found that as the particles begin to reach the larger desired size, coagulation occurs. Coagulation is caused because the surfactant molecules on the surface of the micelle are stretched too thin to cover the much increased surface area of this larger particle, thereby allowing the monomer swollen polymer particles which are soft and viscous at this stage in the polymerization process to coalesce. The polymer particle size at which this coalescence tends to occur will, of course, depend upon the particular surfactant employed, polymerization temperature, amount of water in the system and similar considerations. In order to overcome the problem of coalescence during particle growth, additional surfactant may be added to the system when the particles begin to reach the size at which coalescence tends to occur. However, the addition of this surfactant must be very carefully controlled so that new micelles are not formed in the system because these would provide new polymerization sites and would thereby tend to destroy the particle size control established to that point in the polymerization. This may be accomplished by adding the particular surfactant at a rate such that the concentration of free surfactant not yet added to any one of the growing micelles is below the critical micelle concentration for that particular surfactant. The critical micelle concentration is defined as that concentration of surfactant at which new micelles will begin to form and this figure is available for each particular surfactant from the manufacturer. For example, the critical micelle concentrations of aerosol OT and aerosol MA, which are sodium dioctyl sulfosuccinate and sodium dihexyl sulfosuccinate respectively, are 0.07 grams per hundred milliliters of water at 25°C. and 0.6 grams per hundred milliliters of water at 25°C. respectively. Any suitable surfactant of the well-known cationic, anionic and non-ionic types may be employed. Typical examples of surfactants are naphthalenic soaps or sulfonates; sulfonation products of glycerides or their fatty acids; sulfonated derivatives of esters of mono- and polyvalent

alcohols; esterification products of fatty acid and sulfonated monovalent alcohols; sulfonated derivatives of fatty acid esters, such as the diamyl or dioctyl esters of sodium sulfosuccinic acid; sulfonation products of fatty amides; ketones, aldehydes, and nitriles; sulfonation products of natural and synthetic alcohols; such as alkyl sulfates and sulfonates, phosphoric and pyrophosphoric esters of fatty alcohols, such as the sodium salt of 2-ethyl hexyl phosphate; amino carboxylic acids; sulfonated aromatic hydrocarbons, such as the sodium alkyl aryl sulfates and sulfonates, alkylated aryl sulfonates; sulfonates and chlorosulfonated paraffin hydrocarbons; sulfated and sulfonated derivatives of nonionic compounds such as the sodium alkyl aryl polyether sulfates and sulfonates; aliphatic amines and their derivatives, aromatic amines having fatty chains; fatty amines of aliphatic diamines; quaternary ammonium compounds (bases and halides), such as dimethyl phenyl benzyl ammonium chloride, decyl trimethyl ammonium chloride, dodecyl trimethyl ammonium chloride, tetradecyl trimethyl ammonium chloride, hexadecyl trimethyl ammonium chloride, octadecyl trimethyl ammonium chlorides amides derived from amino alcohols and their quaternary ammonium derivatives; basic compounds of pyridinium and its derivatives; polypropanol and polyethanol amines, urethanes or basic salts of ethylene diamine; condensation products of fatty substances and their derivatives with ethylene oxide, such as the polyoxalkylene ethers of partial lauric, palmitic, stearic, or oleic acid esters; and condensation products of phenolic compounds having side chains with ethylene oxide such as the condensation product of dodecyl phenol with ethylene oxide. It also may be desirable in some instances to employ two or more surfactants to optimize properties of the emulsion such as stability, micelle control and particle growth.

Any suitable system of catalysts or initiation may be used in the polymerization process, including, for example, free radical catalysts with a peroxide, a persulfate, an azo compound, oxygen or ultraviolet light. With a catalyst such as a peroxide or a persulfate such as ammonium persulfate, the redox form of free radical catalysts in which a water soluble reducing agent such as sodium sulfite, sodium bisulfite or ferrosulfate is included may also be employed so as to allow for more rapid polymerization at relatively lower temperatures.

Any suitable vinyl-type monomer may be used in the process of this invention. Vinyl-type monomers are defined as those monomers containing the characteristic ethylenically unsaturated structure: $>C=C<$, and capable of undergoing addition polymerization. Typical vinyl-type monomers include;

ethyl maleate, and mixtures thereof; vinyl and vinylidene halides such as vinyl chloride, vinyl fluoride, tetrafluoroethylene, chlorotrifluoroethylene, and mixtures thereof; unsaturated aromatic compounds such as styrene and alkyl styrenes such as alpha-methyl styrene, parachlorostyrene, paramethoxystyrene, and mixtures thereof; unsaturated amides such as acrylamide, methacrylamide and mixtures thereof; unsaturated nitriles such as acrylonitrile, methacrylonitrile, haloacrylonitrile, phenylacrylonitrile, vinylidene cyanade, and mixtures thereof; N-substituted unsaturated amides such as N, N-dimethyl acrylamide, N-methyl acrylamide and mixtures thereof; conjugated dienes such as butadiene, isoprene and mixtures thereof; unsaturated ethers such as divinyl ether, diallyl ether, vinyl alkyl ether and mixtures thereof; unsaturated ketones such as divinyl ketone, vinyl alkyl ketone and mixtures thereof; unsaturated aldehydes and acetals such as acrolein and its acetals, methacrolein and its acetals, and mixtures thereof; unsaturated heterocyclic compounds such as vinyl pyridine, vinyl furan, vinyl coumarone, N-vinyl carbazole, and mixtures thereof; unsaturated alicyclic compounds such as vinyl-cyclopentane, vinyl-cyclohexane and mixtures thereof; unsaturated thio compounds, such as vinyl thioethers, unsaturated hydrocarbons such as ethylene, propylene, polymerizable hydrocarbon fractions, isobutylene and mixtures thereof; allyl compounds such as allyl alcohol, allyl esters, diallyl phthalate, triallyl cyanurate and mixtures thereof.

Any suitable mixture, copolymer or terpolymer of the above vinyl-type monomers may be used in the process of this invention.

Once the polymer particles have been grown to the desired size in the aqueous phase, any suitable coloring technique may be employed to impart the desired color to the polymer particles prior to drying the aqueous liquor from the system. For example, any suitable water soluble dye may be dissolved directly in the latex prior to drying so that a film of this dye will be formed on the particles as the water evaporates. Typical water soluble dyes include, for example, anthraquinone type — C. I. Acid Blue 127, triphenylmethane type — C. I. Acid Blue 103, azine type — C. I. Acid Blue 98, xanthene type — C. I. Acid Violet 9, nitros type — C. I. Acid Green 1, monoazo type — C. I. Acid Yellow, diazo type — C. I. Acid Green 20, xanthene type — C. I. Acid Red 92, quinoline type — C. I. Acid Yellow 3, diazo type — C. I. Acid Orange 79; basic dyes, such as for example, thiazole type — C. I. Basic Yellow 1, ketone imine type — C. I. Basic Yellow 2, acridine type —

C. I. Basic Yellow 4. In another technique the polymer particles in the latex may be dyed with disperse dyes which generally come from the water insoluble azo or anthraquinone classes of dyes. These dyes are dispersed in the aqueous phase of the latex with a surfactant from which the dyes are believed to enter the polymer particles in the form of a solid solution. Typical disperse dyes include, for example, nitroacetamine Yellow 2 RZ — C. I. Disperse Yellow 1, azo — C. I. Disperse Orange 3, azo — Disperse Red 1, anthraquinone — C. I. Disperse Violet Celanthrene Red, anthraquinone C. I. Disperse Blue 9, amino ketone — C. I. Disperse Green 1, azo — C. I. Disperse Black 9, C. I. Black 18, 19, 16, 1, 7, 12, 24 and 27; and diazo — C. I. Food Black 1. Any other suitable dye may be used if desired.

Once the desired color has been imparted to the polymer particles, the liquid portion of the emulsion is removed by spray drying or any other suitable drying technique which will not cause agglomeration or coagulation of the colored polymer particles. The dried particles are then collected and are ready for use in the electrostatographic process. Although heat may be employed in the drying process, care should be taken to avoid heating the polymer particles above their caking temperature so they will remain as discrete particles. Spray drying has been found to be a particularly preferred technique because it produces fast elimination of the aqueous phase without causing agglomeration of the polymer particles. Since the polymers used for electrostatographic purposes will not form films as they come out of the latex and will not soften until they reach a point well above ordinary ambient temperatures, moderate heating during drying presents no problem.

In the following examples, which will more specifically define the present process in relation to various preferred embodiments, all parts and percentages are by weight unless otherwise specified. In these examples, the drying apparatus employed is a Bowen laboratory size spray dryer manufactured by Bowen Engineering, Inc., North Branch, New Jersey. This spray drying unit is a laboratory size conical spray dryer with concurrent air flow and has an interchangeable atomizing head mounted near the top of the drying chamber and fitted inside the drying air distributor ring. Any one of a number of well-known atomizing devices as commonly employed in spray drying apparatus may be employed with the dryer, such as centrifugal or swirl-type pressure nozzles, pneumatic or two-fluid atomizing nozzles in which a jet of the liquid is disintegrated as it is struck by a nozzle by a high velocity gas stream, ultra-

sonic nozzles in which atomization is accomplished by supersonic vibrations impinging on a liquid stream or atomization by impingement of the stream against a solid surface. Unless specified, the atomizer used in the following examples was of the spinning disc-type in which the liquid is broken up by discharging it at a high velocity from the periphery of a rapidly rotating disc. A pneumatic-type nozzle was also successfully employed. The spinning disc atomizer used employs a disc having a radius of 1" and was operated at 50,000 RPM's. Once the liquid has been atomized, it moves through the drying air until the water is driven off by evaporation which is hastened by the high surface-to-mass ratio of the droplets. The time during which these droplets are held in suspension in the drying air is referred to as the dwell time. With most polymers which are of interest for electrostatographic uses, the drying air may be heated moderately to hasten drying so long as this heat is maintained below the caking temperature of the polymer. This heating may come closer to the softening point of the polymer than might be expected because evaporation of the water from the droplets tends to cool the particles to some extent. In the following examples, the drying air input temperature is maintained at 160°F. and this will generally produce a measured drying air output temperature of about 135°F. The output of the spray dryer is connected to a cyclone-type product collector from which the final dried particles may be taken at the end of the process. A toner made according to this technique was tested in a conventional selenium plate-type xerographic apparatus and found to produce a very good image.

EXAMPLE I

A monodisperse polymer is prepared by a process which includes preparing an aqueous and monomer phase separately. The aqueous phase comprises about 100 ml. of deionized water, 0.5 grams of IGEPON T-51 (sodium N-methyl-N-oleoyl-taurate) OCH_2 , $\text{C}_7\text{H}_{15}\text{C-N-CH}_2\text{-CH}_2\text{-SO}_3\text{Na}$) and 0.3 grams of $\text{K}_2\text{S}_2\text{O}_8$, agitated to form surfactant micelles. In the monomer phase about 50 ml. of the comonomers are mixed with about 0.5 grams of Aerosol OT-100 (sodium dioctyl sulfosuccinate). The comonomers comprise about 65% styrene and about 35% n-butyl methacrylate; this composition is referred to below as 65/35 Monomer Mix. The emulsion is made by blending these two phases with an ultrasonic mixer or other suitable means of mixing until the monomer phase is completely dispersed in the aqueous phase. This emulsion is then placed in a glass vessel and sealed. The vessel is placed

in an oil bath maintained at a temperature of about 70°C. and rotated end over end.

The second and succeeding step in the preparation of a monodispersed latex is based on increasing the size of the particles by a seeding technique. This seeding technique consists of adding the charge of latex prepared as above to a new vessel containing deionized water and dissolved potassium persulfate ($\text{K}_2\text{S}_2\text{O}_8$). The contents are then shaken until completely mixed. To the aqueous phase a predetermined charge of monomer mix is floated on top of the diluted latex with no mixing of phases at this point. Polymerization of the latex is carried out by polymerizing in an oil bath maintained at about 70°C. or other appropriate temperatures using end over end mixing. This procedure is then repeated until surfactant encapsulated copolymer particles of the desired degree of monodispersity are obtained. The following table indicates the procedure to be used in preparing a monodispersed copolymer with a mean particle diameter of 2 microns.

Latex No.	1	2	3	4	5	6	7
Seeded with	none	1	2	3	4	5	6
ml. Seed	none	30	30	30	30	30	30
Latex	none	30	30	30	30	30	30
65/35							
Monomer Mix (ml)	50	40	40	40	40	40	40
g. $\text{K}_2\text{S}_2\text{O}_8$	0.3	0.3	0.3	0.3	0.3	0.3	0.3
Ml. H_2O	100	80	80	80	80	80	80
g. Aerosol OT-100	0.5	—	—	—	—	—	—
g. IGEPON T-51	0.5	—	—	—	—	—	—

Latex No. 7 in the above table will produce a monodisperse latex with a mean particle diameter of about 2 microns. Using the same technique for growth, particles may be formed in the 4 to 5 micron range by increasing the amount of monomer mix to give a monomer-to-polymer weight ratio of about 15 to 1.

After the emulsion is grown to the desired point, it is diluted with deionized water to about 5% solids. A 10% Eosine bluish dye is then added to the emulsion and mixed with a Bronson Sonifier ultrasonic mixer to insure complete dispersion prior to drying. The resulting liquid mix is then spray dried using a pneumatic nozzle at about 100 psi.

EXAMPLE II

The same procedure was followed in this example as is described in the first step of Example I. The monomer phase in this example comprises 120 grams of a monomer comprising 35% n-butyl methacrylate and 65% styrene and about 4 grams of Aerosol OT-100. The aqueous phase comprises about 400 ml. of deionized water, one

gram of ammonium persulfate and about 0.56 grams of sodium sulfite. The emulsion is then made by blending these two phases until the monomer phase is completely dispersed in the aqueous phase. This addition is carried out in a suitable vessel and the polymerization carried out at about 40°C. for about 20 hours. In this case, sufficient monomer is included in the initial formulation so that no reseedling is required to produce the monodisperse, surfactant evaporated copolymer particles with a mean particle diameter of one micron. After polymerization has occurred, 0.5 grams of acridine yellow dye is dissolved in 20 ml. of deionized water. About 20 ml. of the above produced latex is added dropwise to the aqueous solution with stirring. The latex is then spray dried to form a yellow toner. The toner is then used in a xerographic process and gives excellent quality prints and high resolution.

In Examples III and IV, the procedure of Examples I and II is followed except that other dyes were used such as Naphthol Blue Black and C. I. acid blue 127.

EXAMPLE V

0.5 grams of IGEPON T-51 surfactant and 0.3 grams of $K_2S_2O_8$ initiator is added to one liter of water in a pressure reactor and agitated to form surfactant micelles. The reactor is then heated to 45°C. and connected to a gas cylinder containing vinyl chloride monomer. 500 grams of monomer are slowly metered into the reactor while the aqueous phase is maintained in continuous agitation. The monomer slowly diffuses into the aqueous phase and polymerizes into a polymer of uniform particle size. Monodisperse, surfactant encapsulated polymer particles with a mean particle diameter of about 1 micron are produced. At the conclusion of the polymerization, 50 grams of Eosine bluish dye are added to the emulsion so as to color the polymer particles. The emulsion is then spray dried to produce the toner particles.

EXAMPLE VI

0.5 grams of IGEPON T-51 surfactant and 0.3 grams of $K_2S_2O_8$ are added to one liter of water in a pressure reactor and agitated to form surfactant micelles. To this water phase there is added a liquid monomer phase consisting of 400 ml. of styrene and 5 grams of AEROSOL OT-100, (available from American Cyanamid Company). Addition of the monomer phase is made during the maintenance of continuous agitation in the reactor. The reactor is then heated to 45°C. and connected to a gas cylinder containing butadiene monomer. 100 grams of the butadiene are slowly metered into the reaction vessel during continuous agitation, so that monodisperse surfactant encapsulated polymer particles with a mean particle diameter

of about 0.8 microns in diameter are formed. At the conclusion of the polymerization, 50 grams of Eosine bluish dye are added to the emulsion to color the polymer particles and the emulsion is spray dried to separate the particles from the aqueous phase.

WHAT WE CLAIM IS:—

1. A method of preparing electrostatic toner particles comprising forming a plurality of surfactant micelles in an aqueous medium and providing in said aqueous medium, in the presence of a water soluble polymerization catalyst, a sufficient supply of at least one ethylenically unsaturated monomer to form an aqueous suspension of monodisperse, surfactant encapsulated, polymer particles having an average size from about 0.01 to about 50 microns in diameter in said aqueous medium by preferential diffusion of monomer to the smallest of said polymer particles during polymerization, adding to said aqueous suspension a dye capable of colouring said polymer particles therein and then separating said particles from said aqueous suspension.
2. A method according to claim 1 wherein a plurality of surfactant micelles is formed in said aqueous medium before adding said monomer to said aqueous medium.
3. A method according to claim 1 or claim 2 wherein the step of adding a dye to said aqueous suspension comprises adding a water soluble dye thereto.
4. A method according to claim 1 or claim 2 wherein said step of adding a dye to said aqueous suspension comprises adding a dispersed dye and a surfactant thereto.
5. A method according to any of claims 1 to 4 wherein the step of separating said polymer particles from said aqueous suspension comprises spray drying said suspension to remove said aqueous medium by evaporation.
6. A method according to any of claims 1 to 5 in which said polymer particles have an average size ranging from about .01 microns to about 10 microns in diameter.
7. A method according to any of claims 1 to 6 wherein said monomer is continuously supplied to said aqueous medium.
8. A method according to any of claims 1 to 6 wherein said monomer is intermittently supplied to said aqueous medium.
9. A method according to any of claims 1 to 6 wherein said supply of monomer is all added initially to said aqueous medium.
10. A method according to any of claims 1 to 9 wherein surfactant is added during said polymer particles, the addition of surfactant being so controlled as to maintain the concentration of free surfactant below the critical micelle concentration as hereinbefore defined.
11. Electrostatic toner particles

produced by the method of any of claims 1 to 10.

12. A method of preparing electrostatic toner particles according to claim 1 substantially as herein described.

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